# LEAD PLATING: ANU SLRS UPGRADE

N.R. Lobanov<sup>#</sup> and D. W. Weisser, Nuclear Physics Department, Research School of Physical Sciences and Engineering, Australian National University, ACT, Canberra 0200, AUSTRALIA

### Abstract

ANU has adopted MSA chemistry to replate the first SLR in June 1999. This is motivated by the proven success of lead plating at Stony Brook. The entire ANU Linac (10 SLRs at average field of 2 MV/m, lead plated in Oxford) will be replated subsequently. Surface preparation and repair of the crack in electron-beam weld with low temperature solder are described. A lead plating process on samples and on SLRs will be outlined. The composition of impurities stemming from the plating process will be discussed.

#### 1 INTRODUCTION: LEAD ON COPPER

Lead plating was recognized as an appropriate technique providing just slightly poorer performance (BCS losses) compared to a Nb superconducting layer at low frequency. Lead plating provides fast moderate results with modest equipment and relatively low cost. In order to minimize lead oxidation and contamination during post-plating treatment, lead-tin was adopted for SUNY QWRs and the Munich cyclotron. Pb<sub>0.96</sub>Sn<sub>0.04</sub> has a critical temperature of T<sub>=</sub>7.5 K. In spite of the reported difficulties in controlling the concentration and lower RRR value of tin-lead compare to pure lead (50 for pure lead compare to 24 for 2% tin-lead), both laboratories demonstrated good results. Today the "traditional" resonator plating chemistry is commercially no longer available because both Shinol and fluoborates have become obsolete for economic and environmental reasons. SUNY replated six high-beta SLRs with 2 microns of Pb-Sn alloy using a modern, commercial process, LeaRonal Solderon, and a simple open-air procedure [1]. While MHS-L is the only LeaRonal additive suitable for resonator plating, there are similar MSA products available from other companies that might work as well. The new plating chemistry and simple techniques have proven remarkably successful for three reasons: 1) consistently superior superconducting performance; 2) ease and speed of application; 3) greater safety and environmental compatibility. The improved performance at SUNY makes possible routine operating fields 10-20% higher than the previous 3.0 MV/m. Two of their six resonators can be operated at accelerating gradient of 4.0 MV/m for power dissipation of 10 Watts. In addition, SUNY completed the QWR upgrade of the

low beta section. Noé presented an excellent overview on the latest developments in lead plating technology in [1a, 1b]. Many questions about MSA-based leadcopper technology remain unanswered dependence of superconducting performance on tin content, film thickness, surface activation, additive concentration, metal impurities and distribution of RF power loss on the SLR surface. Nevertheless, the proven success motivated ANU to adopt MSA chemistry and to replate the first SLR in June 1999 to be followed by replating the entire ANU Linac (10 SLRs at average field of 2 MV/m, lead plated in Oxford). This might increase the booster energy gain with the same numbers of resonators by up to 100% if the best SUNY results is achieved or 50% if the average SUNY results are obtained.

LNL continues using the traditional fluoborate bath and is producing of home made Shinol. LNL experience in lead plating is supported by the high number (120) of successful plating cycles performed so far and by sixty working resonators produced for the Linac [2]. Quality testing, during mass production, was applied to only 15% of total number of QWRs based on good reliability of the plating process. Recent developments have addressed post-plating surface passivation [3] consisting of careful rinsing of the job with pure de-ionized water with intermediate immersion in light acid solution. The acid dissolves oxides and modifies the lead surface. The goal is to remove all traces of fluoborate by means of subsequent dilutions.

In 1998, LNL reported that four lead plated QWRs from ALPI were taken off-line because two of their couplers failed. New cavities have a new coupler design, making them independent of the tuner mechanics. The Nb sputtering was trialed as a substitute for the electroplated lead coating in four medium beta resonators. This required minor changes in the resonator shape. Fields in excess of 4.5 MV/m at 7 Watts could be obtained off line even though the cavity geometry was not optimized for the sputtering procedure [4].

If total replacement of the installed medium beta lead plated QWRs with sputtered units is adopted, then significant improvement in ALPI performance and

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<sup>#</sup> E-mail: Nikolai.Lobanov@anu.edu.au

reduced the cryogenic power consumption are possible.

Further development Nb technology probably will make it much more attractive than lead. In spite of impressive results achieved so far with lead plating, the Nb technology offers better material with better RF performance, better stability on being exposing to air and environment friendly "dry" deposition technique. Nevertheless lead plating is still the dominant process for substrates with complex geometries like SLRs.

### 2 PLATING BATH

Methanesulfonic acid (MSA) is used for lead plating. It is less corrosive and toxic than fluoboric acid. The LeaRonal in 1984 introduced additive MHS-L (Matte-High-Speed-Lead) for pure lead with little tin. There are similar MSA products available from several other companies for example Technic in USA and Schlotter in Germany.

The metal, the electrolyte and various other deposition conditions determine the growth type of an electrodeposit. deposition is accompanied by Cathodic metal simultaneous hydrogen deposition. The lead having the highest hydrogen overvoltage among other metals does not absorb hydrogen. Hydrogen deposition is responsible for the formation of pores in electrodeposits [5]. The deposition of lead-tin is one of the simplest alloy plating system since the electromotive potential of the two metals differ by only 10 mV. The potentials of lead and tin are only slightly less noble than hydrogen and, since the metals have a high hydrogen overvoltage, the alloys that deposit out of the acid solutions have a cathode current efficiency of 100%. On the basis of microscopic examination, low melting point metals such as lead and tin are field-oriented deposits when single crystals are formed in the electrodeposits. These can be very large and often have dendrite branching with the consequence that continuous coatings are generally not formed. The addition of additive eliminates this behavior. Bright deposits are characterized by their large number of lattice defects due to the incorporation of impurities, such as foreign metal ions or organic species, into the electrodeposit. The impurities or brighteners, which are codeposited with the metal cations, are thought to function by inhibiting crystal growth parallel to the substrate surface such that there tends to be preferential growth in the direction of the current or the prevailing electrical fields. The defect structure of bright electrodeposits results in a tendency to high internal stress and brittleness. In contrast, matte or dull deposits have fewer defects and are generally purer in composition [6]. With a well functioning plating MSA bath, a copper substrate can give highly uniformed fine-grained matte or satin appearance which is associated with the best operating performance [1].

The split loop resonator body (a 40 cm dia by 22 cm long copper cylinder with internal loops) and two tuning plates are plated separately. The entire resonator, or tuning plates is immersed in the 80-liter plating bath containing: MSA 35% by volume (28 liters); lead concentrate 5% (4 liters); tin concentrate 0.5% (0.4 liters); MHS-L additive 5% (4 liters) and DI water 55% (44 liters). The low metal content is appropriate where high deposition rate is not an issue and simplifies disposal. As was mentioned above, the MSA bath has very high current efficiency  $\eta$  (up to 100%), so secondary chemical reactions do not influence the quality of the coating. The adopted 0.5% tin was tested at LeaRonal on samples and corresponds to about 4 atom % tin in the deposit [1]. Only half amount of MHS-L was used without problem, 5% compare to 10% in the current LeaRonal technical specification.

The plating solution was made up directly in the plating tank after leaching the tank and associated pipes, pump and filter overnight with a 10% MSA solution [7]. Proper filtration is important for a good deposit. Continuos filtration with the fluid passing four times per hour is sufficient. A satisfactory alternation is batch filtration before plating of about ten times the solution volume. The filter is one micron polypropylene cartridge. In order to reduce ageing effect and solution contamination, after use the bath is pumped into previously leached HDPE sealed containers with dry nitrogen gas above the liquid. These are equipped with pressure relief valves at 2-3 PSIG. While stored in the open plating tank, dry gas nitrogen flows continuous above solution level. When filtering or pumping the solution, care is taken to avoid entrapping air. During periods of shut down, the solution is protected from the atmosphere and is filtered before resuming operations to remove any stannic tin that may have formed. With time a fine light brown precipitate develops in the solution. This could be removed by adding a coagulating agent LeaRonal Clarostan before filtering [1].

The anode is made of lead sheet 0.5 mm thick with purity of 99.99%. The small quantity of lead reduces the cost of disposal. However, the sharp edges of the thin anode enhance the electric field. In order to reduce field enhancement  $\beta$  and associated excessive current density, the edges of the anode sheet are bent to a radius of about 2 mm. Based on data for field enhancement from [8a] this reduces  $\beta$  from 300 to acceptable value of 30. The anodes are removed from the bath between plating. A two micron thick film in the SLR with the surface area of 0.63 m² contains about 14 grams of lead. The plating bath contains 1.8 kg of lead. If one assumes all plated lead is replaced from the anode, only 0.01% of anode impurity enters the plating solution during each process.

The effects of metallic contaminants in the anode were studied in [8b] as they affected the Pb/Sn fluoborate plating process. It was discovered that out of the impurities studied in the anodes (Ag, As, Au, Bi, Cd, Cu, Fe, In, Ni), no effects were noted on the quality of the electrodeposits. The solution impurity levels were not affected in any significant manner by any anode impurities, thereby resulting in no co-deposition. Analyses of the plating solutions, and of the sludges produced, confirmed that the anode impurities studied were left behind in the anode sludges. The lack of impurity carryover from anode to plating solution is probably due to the fact that, in the anode, most of the impurities are present in the form of intermetallic compounds which are very resistant to chemical dissolution.

The solution impurities investigated in [8b] were cadmium, copper, iron, nickel and zinc. These were studied because it was thought that they would be the most likely to find their way into the plating bath through contact with hardware, fixtures and substrates. Copper was found to be the only impurity that had a great overall influence on the process. Besides being the only impurity to co-deposit with the tin and lead, it also influenced the colour of deposit, the amount of occluded organics in the deposit and also the corrosion of the anode.

The anode sheets cover both sides of the plating tank perpendicular to the resonator axis and are visible from anywhere inside the resonator. The total surface area of two anode sheets of 4x0.4x0.4=0.64 m<sup>2</sup> provides anode to cathode ratio of 1:1 [9] thus excessive current density is prevented. Napped polypropylene cloth bags cover the anode to prevent solid particles and debris from getting into the plating bath and causing rough deposits. Basic lead carbonate (PbCO<sub>3</sub>), Pb(OH), and lead oxides PbO and PbO, are formed where there is atmospheric exposure. The anode should be free of oxides and nonmetallic inclusions and low in metallic impurities. Prior to installation in the plating bath, the lead foil is cleaned in vapour degreaser, acetone and methanol following by high pressure rinse (HPR). Immersion in the 5% EDTA solution for 2-3 hours following by rinsing in DIW completely removes all the lead corrosion products from lead anode foil. No sign of carbon contamination in the plated lead was found by SEM technique though this technique might miss thin surface stains. The dc power is supplied by means of copper bus bar, which is positioned above the tank and runs three-quarters the length of the tank. The anodes are suspended from the bus bar. The SLR is attached directly to the cathode forming twodirectional pneumatic arm. The arm may move SLR in vertical and horizontal direction.

## **3 CRACK REPAIR**

CALTECH Low Temperature Physics, in conjunction with the Nuclear Physics Department at Stonybrook, developed  $\beta$ =0.065 and  $\beta$ =0.10 SLRs for the Stonybrook Linac. SLRs were then manufactured by ASI, the first one of which, labeled "300", was bought by ANU for a super-buncher [10]. Shortly after the ANU order, a 10 resonator Linac was bought by the Nuclear Physics Department at Oxford University. The ten SLRs are numbered 301 to 310. Earlier in Oxford upon inspection and leak-testing, three of them (301,304 and 308) were found to have vacuum leaks, due to failed electron beam welding at the base of the stem that supports the split ring (see figure 1 and figure 2).

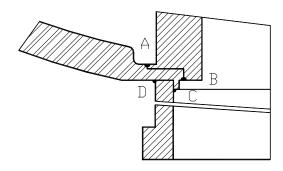


Figure 1: The electron-beam weld of the  $\beta$ =0.10 SLRs



Figure 2: Photograph of cracks and exposed copper in electron-beam weld of resonator 304

The resonators were shipped to the USA for repairs and were returned to Oxford in 1985. In 1987 it was found that cavities 304 and 308 were still leaking in spite of two successive welds. It was suggested that a normal post welding leak check on these resonators in the USA would not have revealed the problem, which became apparent only when the cavities were

subjected to the rigorous cleaning procedure, prior to plating. The cleaning process may have etched trough a too thin weld "A". As well, weld "B" must also not be leak tight. The Special Techniques Group at Culham decided to repair the welds by the technique of local electroplating. The area around the crack was machined flat and a new layer of copper was electroplated to a thickness of 0.5-1 mm. In spite of the delay and the high cost, the technique proved to be successful. Two cavities were thermally cycled in liquid nitrogen by Oxford and vacuum baked, no leaks were found. The equipment eventually found its way to Australia reuniting the first ASI resonator with its long separated siblings.

Unfortunately ANU cavities 300 and 304 exhibited cracked welds, not vacuum leaks though, in November 1998 when they were opened for plating with MSA chemistry. The electron beam weld was exposed in cavity 300 as a result of a few chemical etches during trial plating. SLR 304 developed a few cracks, stain and exposed copper near the weld area (see figure 2).

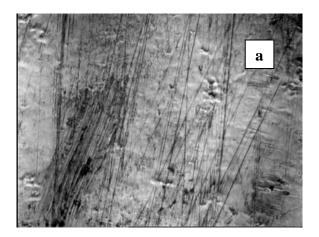
Probably the small crack and porosity existed prior to lead plating. The crack captured plating solution that later caused corrosion. As a result of poor adhesion caused by the corrosion, a few copper areas up to 2 mm diameter were exposed.

The repair of the resonators presented severe technical problems for the following reasons: 1). All available information indicated that any attempt to run, the welding electron beam over the faulty weld, would be counterproductive, resulting in re-crystallization of the copper and the possibility of further cracks; 2). It would be highly desirable to avoid heating the resonator to more than 200 °C for brazing or high temperature soldering because this would result in distortion and change of resonant frequency; 3). The internal surfaces under the weld could be contaminated by corrosive materials left after previous plating; 4). The weld area might still be under stress due to high local heating during electron-beam welding. This could cause creation of new cracks in surrounding bulk copper during thermal cycles.

The proposed ANU solution was soldering over the defective weld. The standard 60Sn/40Pb solder was initially rejected because it becomes brittle at cryogenic temperature [11]. Solders with higher lead content remained ductile down to temperatures near absolute zero. However, high-lead solder has too high melting point around 300°C. The fact that the cavity would not be heated in service above 80°C allowed the choice of pure indium or indium alloy as low temperature solder material. Its extreme softness and

pliability make it an ideal soldering material between surfaces experienced high thermal contraction.

This indium alloy was chosen after analyses of the number of solders commonly used for low temperature research [12]. Figure 3 demonstrates change in 60Sn/40Pb and indium solders on copper sample simulating the crack in SLR after 10 thermal cycles in liquid nitrogen. Due to different coefficients of linear expansion of the copper substrate and the solder, cracks and stress lines were developed only in cryogenically brittle lead-tin solder (figure 3a) while indium-based solder remained unstressed (figure 3b).



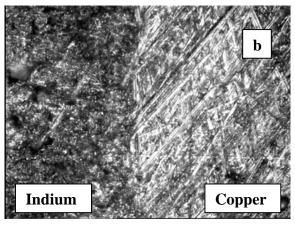


Figure 3: Stress lines and cracks in 60Sn/40Pb (a) and no developing of stress in indium-based solder (b) on copper sample after 10 thermal cycles in liquid nitrogen.

The first soldering trial was done with pure indium as solder material. The mild inorganic salt flux for copper EUTECTOR 157, effective with low temperature solders, was used as a first trial. Its composition is 50 g of zinc chloride, 1 g of sodium fluoride in 10ml of water. The soldering temperature was 20 to 65 °C above the melting point in order to

give the indium good fluidity and wetting characteristics. The post-solder cleaning included a rinse in hot water, neutralizing in 2% HCl solution twice each, followed by rinsing in organic solvent and cleaning in Cuprolite X84C. This was followed by a final rinse in DIW and HPR. Better results were achieved with the flux COMWELD 965 (COMWELD GROUP Pty). This contains an eutectic combination of zinc chloride-ammonium chloride diluted in HCl with melting point at 176 °C. Acetone and water used to facilitate application.

When zinc chloride is a major component of the flux, the following reaction will takes  $ZnCl_2+H_2O=Zn(OH)Cl+HCl$ . HCl is now ready to clean the oxides off the surfaces  $CuO+2HCl=CuCl_2+H_2O$ . Thus the copper chloride, which is very soluble in water, can easily be removed. Unless the zinc oxychloride is removed with a dilute acid (2%HCl)  $Zn(OH)Cl+HCl=ZnCl_2+H_2O$ , a layer of dull white may remain in the vicinity of the joint. The restored  $ZnCl_2$  is water-soluble.

In order to eliminate chemical corrosion, the flux residues and fumes have to be totally removed from the assembly. The importance of the adequate removal of chlorides from copper covered by lead film is illustrated as following [11]. A dense well-adhering tenacious layer of lead oxides protects the metallic lead film. In the presence of chloride ions the reactions  $Pb(Metal) + 0.5O_{3}(Air) = PbO$ PbO+2HCl=PbCl<sub>2</sub>+H<sub>2</sub>O proceeds. The lead chloride PbCl, is a loosely adhering compound and is easily converted into the more stable lead carbonate  $PbCl_2+H_2O+CO_3=PbCO_3+2HCl$ , resulting in the production of two chloride ions, which are free to attack the lead oxide layer again. The lead carbonate PbCO<sub>3</sub> is porous white layer that does not protect metallic lead. Oxygen reaches metallic lead and there is renewed oxidation.

After receiving Research Solder Kit from Indium Corporation of America, different indium alloy wires Indalloy #1E, #290, #2 and #4 were tested together with low temperature fluxes Indalloy #4, #5RMA and 5R. The best results were obtained with wire #1E and flux #4 recommended for oxidised copper. The composition of wire #1E is 52% of indium and 48% of tin with melting point of 118 °C. Flux #4 is organic salts in water-soluble solvents. In the dual step flux removal process the solder joint is first cleaned using DIW rinse at 40-60 °C. The assembly is then immersed in DECON90 following by HPR.

Sample tests showed soldering could be done with indium alloy melted onto OHFC in air without heavy copper oxidation. Colors of residues appearing on a soldered assembly are used as an identification of corrosion mechanism, if any. White residues may indicate the presence of chloride-containing salts, which continuously react with indium, lead or copper. However a low contact angle between solder and substrate could not be obtained for any combination of indium-based alloys and fluxes from Indalloy Research kit. The 60Sn/40Pb solder with melting temperature of 180 °C was considered again. This solder demonstrated a minimal contact angle and the best wetting of the copper substrate. It is anticipated that 0.5 mm thick layer of solder above the crack should attenuate the maximum stress on working area during contraction due to cooling.

Following these tests, cavities 300 and 304 were processed with 60Sn/40Pb alloy. They contained circular grooves from cracked weld of about 0.5 mm wide and 20 mm long. Using pre-applied flux into the groove the job was heated to 200°C in 35 minutes using 2 kW heater tape outside outer wall and heat gun blowing hot air inside the stub. The temperature was monitored by type K thermocouple. Preheat was used to evaporate water and melt solid flux ingredients to facilitate wetting. Preheating on SLR helped to eliminate mechanical stresses due to localized heating. The uniformed preheating allowed the solder to fill the joint more quickly with a minimum metallurgical reactions and lack of voids. The solder wetted and filled the groove satisfactorily indicating a small dihedral angle, its surface was bright and smooth with no pinholes. The copper cavity during this trial had not been subjected to high temperature so the variation of the critical dimensions of the cavity due to stress relief was not an issue.

Using the general rule for the electronic industry, the maximum service temperature for a particular alloy [11] is given by equation:

$$T_{\text{max}} = 0.67(T_{\text{al}} - T_{\text{room}}) + T_{\text{room}}$$
 , (1)

Where  $T_{al}$  is the melting point of alloy,  $T_{room}$  is the ambient temperature, 18°C. For 60Sn/40Pb solder, which has a melting point of 180°C, the recommended maximum solder temperature in service would be  $T_{max}$ = 127°C, higher than SLR baking temperature of 70°C.

Lead plating of the soldered SLRs presented the following technical challenges. First, the plating bath vigorously attacks tin-lead solder. Second, being in contact with the more electronegative copper, the tin and lead could have constituted a sacrificial anode in a Cu-In galvanic corrosion cell. Electrolytic action between surface areas of different potential causes

corrosion process. For pure metals and single-phase alloys a potential difference exists between grains with different orientations, between grain boundaries and impurity phases. For multiphase alloys a potential also exists between the various phases present, altering the rate of attack. In an electrolyte, the more electropositive phase is attacked while the more electronegative phase is not attacked appreciably. The electromotive potential between copper and tin is 480 mV while between tin and lead 10 mV. Tin, the metal with the higher electromotive potential (140 mV) is sacrificially consumed in this corrosion mechanism. Finally, it is not obvious the lead will be deposited uniformly onto non-uniform copper-tin-lead substrate.

For example in reference [12] the cracks in QWR were covered with indium-silver solder. The lead plated over the solder and the low field Q was measured to be  $4x10^8$ . With increasing power, however, the Q suddenly dropped. The authors contributed this drop to the poor heat transfer properties of solder. Our calculations of heat transfer QuickField confirm that the conductivity of indium-silver solders covering 0.5 mm wide crack is more than adequate to provide the thermal stabilization. In our opinion Q degradation appears to be due to the growth of normal regions in the superconducting surface that are nucleated by localized energy dissipation by surface defects (~1 mm) inherent in the solder found by SEM as areas with high indium concentration. During 4 K operation, once normal regions are nucleated, they can propagate through the lead layer due to the relatively low thermal conductivity of the selected indium alloys (~ 0.43-0.86 Wcm<sup>-1</sup>k<sup>-1</sup>). The same phenomenon was reported in [13] for lead plating over silver brazed superconducting joint, where at field level of only 100 G about 40% of the area over the solder joint appears to be normal corresponding to an average surface resistance of  $4.4 \times 10^{-4} \Omega$ .

Electroplating layers nickel and copper over solder made significant improvement in the joint. When using the brush plating technique, copper substrate and solder area were prepared for as following [14]:

- Pre-clean part. Degrease with solvent and remove oxides, scale, corrosion and organic coatings;
- Electro-clean with sodium hydroxide LDC-01 forward positive polarity 10-15 volts 200 mA/cm² for 2-3 minutes;
- Water rinse thoroughly with DIW, HPR;
- Activate with sulfuric acid LDC#1 forward positive polarity 10-12 volts at 180 mA/cm<sup>2</sup> for 1 minute, water rinse, HPR;

- Immediately plate nickel at forward polarity 16-18 volts;
- Water rinse thoroughly with DIW, HPR;
- Immediately plate copper with LDC-2904 at forward polarity 7-8 volts;
- Soak in citric for 1 minute, rinse, HPR;
- Immediately activate in lead plating bath, plate lead:
- This is a wet process. Move quickly between steps.

Copper was plated with alkaline solution LDC-2904 (Metallock Ltd., Australia) made up of copper sulfate with additives by a brush plating technique to a thickness of few microns [14]. For LDC-2904 solution, the amperehour factor is estimated to be 0.0008 µxcm<sup>-2</sup>. With an active brush surface area of 5 cm<sup>2</sup>, a two micron copper film is deposited during 2.4 minutes at 0.2 A plating current. Activation of the substrate prior brush plating was done by immersion in the 0.5% citric acid for 5 minutes. Equipment was made from materials in the plating shop as recommended in [15]. The plating brush 0.75" wide 3" long was made from platinum. A uniform high purity copper substrate for lead plating across the solder was obtained. Performance throughout the field range up to more than 200 G without noticeable Q degradation due to the soldered cracks is quite similar to that of lead plated SLRs with no cracks. Thus the method provides convenient and economical means of crack repairing for large RF currents. Moreover copper layer over solder protects it against corrosion in MSA plating solution.

### **4 SURFACE FINISHING**

The careful surface treatment of the copper substrate is considered to be the most important step in any coating procedures. A review of available surface cleaning technologies and their applicability to cavity technology is given in [16]. Any defect on the copper surface such as dislocations, grain boundaries or surface roughness will be reproduced by the lead or Nb films [17]. A small defect on the first lead layer will act as a nucleation center for growing a microparticle with poor thermal contact with the copper substrate. The same affect may be caused by inclusion (oxides, sulfides, abrasive, grease, dust or erosion zones due to bubbles [17]) or porosity of the substrate surface [3]. Delayen suggested copper plating before lead plating [18]. Palmieri's group verified that copper plating of the substrate before lead plating considerably reduced the roughness of the plated lead [3]. However, there is a risk of developing more surface defects due to the copper plating. A more appropriate technique to improve the quality of the substrate might be copper sputtering coating.

The surface roughness of the substrate has an influence on surface resistance [19]. Spun cavities provided on average better results than hydroformed cavities, both in term of R<sub>res</sub> and of its increase with RF field. The most apparent difference between the two kinds of substrates is surface roughness. No simple mechanism explains to residual resistance in terms of roughness, although several conditions are possible viz, film crystalline structure, the effective surface area of the substrate, the effective RF path length and greater likelihood of trapping contaminants.

Adhesion on oxide films is generally poor, and such films are usually present on the copper surface although they are not visible to the naked eye. Mechanically polished surfaces are usually not clean and are covered with oxide layers so that deposits on them have poor adhesion. To exhibit good adhesion to the substrate, its surface must be clean, on a microscopic scale, and be free from dirt, oils and grease. With 'clean' substrate the electrodeposit can approach the metal surface to within distances similar to atomic dimensions when atomic bonding occurs. The increased surface area of the rough surface promotes adhesion by 'keying' the deposit. Good deposit adhesion is found with electrodeposits on mechanically polished surfaces, provided all polishing compounds and oxide films are removed. Good deposit adhesion is necessary in RF superconducting devices because the substrate-deposit combination is to withstand thermal and mechanical stresses without any form of separation of the two metals such as blistering or peeling.

In general, the term contamination applies to the following: 1) Particulate matter, including dust, lint, debris and metallic particles; 2) Crystalline materials, that include previous processing solution residues; 3) Coatings of organic materials, including oil, grease, wax and rosin. A cleaning process must have good flushing action to facilitate the removal of particular matter in group 1 above. It also aids in the solution process to remove groups 2 and 3. There are two major groupings of both soils and solvents: 1. Nonpolar materials such as rosins, wax, halogenated and fluorocarbon solvents: 2. Polar materials contain salts, activators, organic and inorganic acids and water as solvent. Since contamination is never pure it ordinary includes both polar and nonpolar ingredients. Thus the cleaning process also requires both solvents.

The surface cleaning of the resonators presented few technical problems, for the following reasons: 1). Available information indicated that cosmetic electron beam welds would be very thin. Any chemical etch would expose more areas; 2). CERN etch solution aggressively attacks lead-tin solder used in cavities 300, 304 and 308 as is shown on figure 4.



Figure 4: Etch of lead-tin solder on copper sample in CERN copper etch solution.

Based on these considerations, the final surface finishing of SLRs was done as follows. First, all cavities were lead stripped in bath 0. LSTRIP2000 was used as lead-tin stripping solution (Chemgraph Pty.Ltd, Australia). A rate of lead-tin stripping was 10 μ/min reducing to 0.5 μ/min for copper substrate. Then selective surfaces of cavities would be thoroughly hand lapped. Subsequent lapping employed a non-embedding garnet GK, United States Products Co with grit sizes from 5 microns to 1 micron in 1 micron decrements. Then all cavities were first wiped clean with solvents and ultrasoft tissues DURX670, Berkshire Corp., then DI-water high pressure rinsed in open air, following by soaking in degreaser DECON90 for 50-60 minutes in bath 1. DECON90 is an emulsion of anionic and non-ionic surface active agents, stabilizing agents, alkalis, non-phosphate detergent builders and sequestering agents. The next step is up to 40 minutes cleaning in vapor degreaser. It employs bromochloromethane nonpolar cleaning solvent with boiling point at 68.1°C. A vapor phase is advantageously used for cleaning because the condensing vapors are pure. They heat the work and soil to speed up the solution process and the "hot" work comes out at the boiling point of the solvent and is dry. Borothene has a very low surface tension so it is unlikely to be pulled into crevices.

Then allowed to soak for 5-10 minute in powerful surface cleaning agent Cuprolite X84C at 65 °C in bath 3. Cuprolite X84C is neutral degreaser-conditioner containing cationic surfactants mixture. The neutral pH prevents attack of tin-lead solder. After the treatment the copper surface shows very

good wetting for a subsequent even micro etching. The manufacture's instructions for rinse temperature should be carefully followed because Cuprolite leaves thin residues over the assembly if not removed correctly. The first water rinse should be done at 40-45 °C to remove all traces of degreaser and this is followed by a second rinse in DIW and HPR. When removing difficult soil, Neutraclean 7 is used. Light organic soils and copper oxides are removed simultaneously. Cleaning action is more rapid at higher temperature, up to 65 °C. The disadvantage of using Neutraclean 7 is more expensive waste management. Then the parts were passivated by soaking in 0.5% citric acid for 40 minutes and rinsed in DIW. The latter step was intended to remove oxides as well as clean the copper. As required for repaired cavities, brush plating onto lead-tin joint was implemented after this step followed by DIW rinse.

Immediately the entire surface of resonator will be electroplated with ~0.5 u of copper in copper sulfate bath. Currently this process is not applied due to lack of appropriate anode material. In [13], the Q curve for lead over plated copper was actually a factor of two better than for the original copper substrate. This is consistent of Judish's result, where a record low R was achieved with a plated copper substrate [30]. An acid copper electroplating solution is a mixture of water, sulfuric acid, copper sulfate and a trace of hydrochloric acid. The cavities kept wet between all operations. Phosphorized deoxidiezed copper (0.04-0.08 phosphorous) is used as anode material sheathed with polypropylene anode bags. Bags are checked for holes or breaks in the bottom seal by filtering with water and allowing to drain. Plating bath is filtered at 1 u level over night prior application. A filtering rate is circulation of 100 liters solution 4 turnovers per hour.

SLR copper plating proceeds as follows:

- The power supply is adjusted so that at its lowest setting, it maintains low current when the SLR is first lowered into the bath. This helps prevent the formation of a low adhesion "electroless" copper layer;
- Lower the SLR into the plating tank until the top edge is at least 2.5 cm below the surface of electrolyte;
- Swish SLR gently and forth to drive any trapped air bubbles off;
- Slowly ramp up the current in 20 seconds to 15 A and plate for 5 minutes. Turn the current up to 30 A and plate for 2.5 minutes to achieve total copper film thickness of about 2 microns;

Remove SLR from the bath and thoroughly rinse
in the rinse tank to remove the most of
electrolyte. Rinse SLR under running DIW to
remove the rest. The water in the primary rinse
tank can be added back into the plating bath to
make up for drag out and evaporative losses.
This is crucial to reducing metals effluent from
process to near zero.

Finally thoroughly HPR again for 10 minute following by immediate immersion in the plating bath while still wet. High pressure water rinsing system was described earlier in [20]. The pressure is directed at 45 to 60° angles in the direction of the oncoming work giving the best results. The spray impingement is obtained by thoroughly designing of shaped nozzle, which generates cone-shaped stream. In contrast to the QWR, in SLR geometry the backup of cleaning liquid in recess and blind areas is small so the cleaning efficiency is high. The liquid is forced into the work, and can penetrate into the tightest spaces even if the surface tension is not ideal. The aim is to force the maximum amount of rapidly moving liquid through the gaps and over the surfaces. The spray waterwetting test was done after each cleaning step described above. If failed, the previous step of the sequence was repeated.

### 5 PB/SN PLATING SLRS

Trial beaker plating in simple geometry systematically tested the surface quality of the copper samples, throwing and covering power and leveling of LeaRonal lead plating bath. The lead deposits were examined by optical microscopic and SEM methods in term of appearance, crystalline structure, adhesion, porosity and corrosion resistance. These tests enabled us to establish appropriate surface cleaning and soldering techniques.

Anode and cathode strips each 4 cm wide and 7 cm high were held parallel and ~5 cm apart in a 5000 ml beaker. All tests were made with 1300 ml of plating solution that has been filtered through fast speed Whatman 54 paper.

Pores were detected by immersing the coated sample in dilute 2% hydrochloric acid. The hydrogen was evolved from acid immersion. The specimen was thermally cycled between 77K and 300K to assess coating adhesion since lifting of the deposit would occur at areas of poor adhesion.

The lead film could not be of uniform thickness because of the irregular profile of SLR. Few properties of the electrolyte exert influence on the character of the film. LeaRonal plating bath demonstrated excellent covering power measured as the depth of penetration of the deposit in cm into a recess of the surface. Macro-throwing power is the expression for differences in the local thickness in the film S=100%(R-M)/(R+M-2), where  $R=D/D_n$  is the ratio of far and near distances between substrates and anode;  $M=M_n/M_f$  is the ratio of amount of lead deposited near and far from anode. For R=2 and M=1.25 the macro-throwing power was estimated to be approximately 60%, slightly above value given in [5]. Micro-throwing power is the property of depositing uniformly into groves, pores and scratches of less than 0.25 mm in width. It is related to leveling and can be expressed as  $E=100\%(d_2-d_1)/t$ , where  $d_2$  is the film thickness in recess;  $d_1$  is the thickness of flat surface; t is the profile depth.

Leveling is produced by MHS-L additive (see figure 5). The additive is constantly being consumed during plating operation at the rate of approximately 0.35 to 0.5 cc per Amp hour. For example, the total plating time for SLR is 7 minutes at plating current of 35 A. This plating job will require about 4 Amp-hours of current and consume about 2 cc of additive. This amount should be added to the bath to insure an optimum operation. The side effect is that the additive is incorporated into the deposit, the amount incorporated decreases with increase in current density. For  $d_2 \cong 4.7 \mu m$ ,  $d_1 \cong 2.0 \mu m$ ,  $t \cong 3.3 \mu m$ , E equals 81% and the rate of deposition is greater in recesses than at the edges of the recesses and this effect arises from the differing degrees of polarization at projections, grooves and pores.



Figure 5: Scratch filling properties of LeaRonal lead plating bath. Two micron lead on a copper substrate.

The throwing power of Solderon plating solution increases with [22b] decrease current density; decrease in temperature; decrease in metal content; increase in free acid content; increase of agitation.

Good macro-throwing power is achieved if the distribution of primary electrical field is uniform and there is sufficient convection within electrolyte to eliminate differences in the thickness of the cathodic diffusion layer. The distribution of the primary

electrical field for a simplified geometry of the lead plating bath with SLR (figure 6) was calculated using code Superfish.

The ratio of peak surface electrical field applied to SLR's outer rim to its minimum value is 22:1 causing an excessive deposition current density. It is imperative that the current densities over all pieces on the job be as uniform as possible. More uniform deposition was achieved by placement the polyethylene ring-shaped diaphragm near the anode opposite to the rim so that the diameter of the diaphragm is 5 cm less than the diameter of the rim.

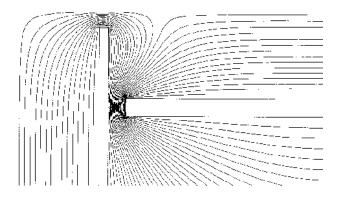


Figure 6. Superfish-distribution of primary electrical field in simplified geometry of plating bath with SLR.

The plating process includes the following major steps (see figure 7). The fifty kilogram pieces are positioned using a custom built telescoping pneumatic hoist on rail-running trolley. The rails are positioned above seven 100 liters vats with chemicals and rinse liquids. The plating bath is placed under the 0.5 micron Hepa-filter, which produces slightly positive pressure and dust free atmosphere for the most important operation. For the same reason, the 25 V 150 A DC power supply is located outside plating area. Manipulation of SLRs and tuner plate is done with self-containing breathing apparatus to prevent personnel exposure to acid mist, ammonia vapors and lead oxide. This also reduces requirements for ventilation of the room.

The plating solution is efficiently filtered through one micron 25 cm long filter cartridge using a self-priming magnetic drive seamless Sethco pump. Polypropylene ball valves BV1-BV5 allow the transfer of plating solution from storage vessels PS to plating vat 4 and back. Lead is plated onto dummy substrate at 10 mA/cm² for twenty minutes through a new bath to clean it up prior to plating the first cavity.

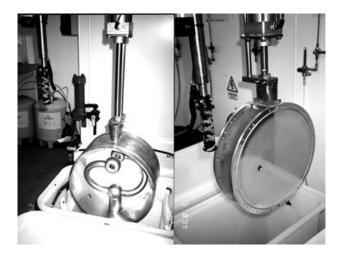
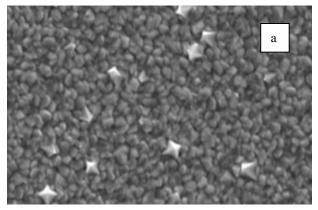


Figure 7: Lead plating process and equipment

Some values important for electroplating can be determined by using the Faraday law [5]. Introducing time t in minutes and current density j in mA/cm<sup>2</sup> we obtain the coating thickness  $d_{\mu} = E_{e} j t \eta / 600 \gamma$ , where  $E_{e}$  is the electrochemical equivalent in g/Ah and  $\gamma$  is density in g/cm<sup>3</sup>. Using this equation for  $E_{el} = 3.865$  g/Ah for lead;  $\eta$ =100%; j=5 mA/cm<sup>2</sup> and t=7 min, the coating thickness of lead is 1.99 µm. All operations of surface preparation and pre-plating copper are described earlier. Activation is done by immersion in the plating bath for 1 minute with low current. This helps to prevent the formation of a low adhesion "electroless" lead/tin layer. Slowly ramp up the current in 20 seconds to 15 Amp and plate for 5 minutes. Steady plating was done for 4 minutes at 5.0 mA/cm<sup>2</sup> (35 A current for SLR and 15 A for tuner plates) for thickness of 2.0 microns corresponding to the best looking surfaces under the electron microscope, figure 8a. From SEM images presented on figures 8a and 8b, the average grain size is proportional to the thickness of lead deposit. Larger grain size should reduce surface resistance. This is supported by the observation [23] as an RRR improvement for DC performance at large thickness. Nevertheless the RF performance indicated the opposite trend. Reference [24a] reported reduction of the maximum RF field with film thickness increasing. This is related to the fact that the surface roughness increases surface resistance due to generation of phonons and causes field emission. Reference [25a] describes a chemical polishing procedure, which is effective on rounding the sharp corners of the 5-10 µ thick polycrystalline lead. However, the best results could not be obtained reproducibly.

The Solderon plating bath plates well at 4-7 mA/cm<sup>2</sup> and forms hydrogen bubbles at over 10 mA/cm<sup>2</sup>. The job was gently agitated (slow cathode movement and circulating through an external pump) to minimize stratification and bubbles formation. Agitation was found to be a crucial

operation when plating SLR repaired with tin-lead solder. However, bath agitation should be mild to keep the formation of stannic tin to a minimum [24b].



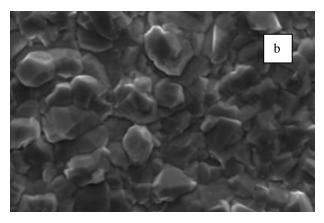


Figure 8: SEM images of lead-tin deposit electroplated onto silica wafer: a) film thickness d=2  $\mu$ ; and b) d=5  $\mu$ . A 0.3  $\mu$  copper layer was e-beam evaporated onto silica prior lead plating.

There are two successive DIW immersion rinses. The first rinse bath 5 is 15 seconds while the second is for about a minute. The second rinse bath contains ~0.015% solution of ammonia, neutralizing bath and lead passivating agent, to bring its pH above 7. Nitrogen bubbled through the bath for 30 minutes to reduce dissolved oxygen. Dissolved oxygen can be also removed by passing the water through a vacuum degassing module that utilizes an inert, gas-permeable membrane surrounded by a vacuum [25b]. As the job was removed from the second rinse bath 6, it was flushed with DIW. The rinse continued until the fresh plated surface did not exhibit a hydrophobic character, which is believed to be caused by a MHS-L additive incorporated into lead film. Deionized water with a resistivity of 4-5 M $\Omega$ ·cm is used for rinsing and for preparing the solutions. Water with a resistivity of  $20M\Omega$  cm has been shown to dissolve thin lead alloys layers [26]. The surface of resonator had satin matte

finish and the few dark stains on the surface if any were easily removed by spraying with weak EDTA solution as recommended in [26]. Optionally spraying with DIW and alcohol was effective.

Drying was done by vigorous blowing with two streams of nitrogen produced by air-knives system. Air-knives consist of GN, supply and nozzles providing wide path through which GN, are dispersed. A GN, line at 80 PSIG powers each of air-knife. The air-knife accomplishes its work in a two-stage process. First, a high velocity, up to 40,000 FPM, stream is directed to roll the liquid back, creating a standing wave at the point of shear and leaving a thin, residual layer. A residual film of liquid dries more quickly than any droplets of the same material. The amount of contaminant in the last rinse stage is smaller in the film than in larger residual liquid pools. Second, this layer is atomized to form minute liquid droplets, which are carried away by the N<sub>2</sub> stream to leave a clean, dry surface. Non-liquids are also blown and removed. Finally, rare isolated drops were lifted from inside surface by gentle touching them with a tissue. Drying was applied to the RF surfaces of the resonator. Large clusters of water from non RF surfaces were collected with tissue following by gentle blowing of remain wet surfaces with low pressure dry nitrogen. As soon as the part was dry it was stored in a nitrogen atmosphere for a few days before assembly.

### 6 ASSEMBLING AND RF TEST

The plated resonators and end plates were assembled in a class 1000 clean room conditions. During assembly, a gentle stream of dry nitrogen directed at the inside of the resonator. A 1.6 mm diameter lead wire seal is used between the resonator body and end plate. The bolts holding down the end plates were tightened down sequentially to torque settings of 7, 11, 12 and 13.6 N·m.

Then the RF field balance is checked. Imbalance in the ring configuration can produce coupling between frequency modes causing net RF current through the lead joint and possibly joint losses [33]. Dimensional tolerances required to reach balance are difficult to maintain during disassembling. For example, the sensitivity of frequency to loop dimensions is typically 140 Hz/µ. Consequently an independent procedure is required to tune the SLR and balance the field profile. Balance was achieved during manufacturing of SLRs by bending slightly each end plate independently. Once balance is achieved the frequency of the resonator can be tuned and maintained upon cooling to low temperature.

Beam extension tubes were attached to each end plate using 1.0 mm diameter indium wire as a seal. The resonator was then attached to the copper pre-cool bar. From this moment and until pump-down time, the

assembled resonator was never left without a bleed of dry nitrogen introduced through the aperture for the pick-up electrode in spite of the fact that no degradation was reported in PbSn coating after four months of exposure to the atmosphere in [27]. The assembled resonators were left to age for about a week prior final room temperature tuning.

The RF surface resistance can be represented as a sum of two terms. First, according to the BCS theory is the temperature dependent part of surface resistance  $R_{BSC_1\Omega_1} \approx 6.85 \times 10^5 f_{[GHz]}^{-1.9} T_{[K]}^{-1} e^{-15.1/T} \approx 12 \text{ n}\Omega$  for  $Pb_{0.96} Sn_{0.04}$  at 4.2 K for 150 MHz [28].  $R_{BSC}(\omega,T)$  decreases strongly with decreasing frequency and temperature. The second contribution, residual resistance R<sub>RES</sub>, is independent of temperature and is proportional to  $\omega^{\alpha}$ . For niobium, residual resistance as low as ~1 n $\Omega$  is reported. However for electroplated lead, to attain low R<sub>RES</sub>, considerable effort in the surface preparation is needed. In [28, 29] the residual resistance of both 1 µ and polished 5 µ thick electroplated Pb<sub>0.95</sub>Sn<sub>0.05</sub> layer in a reentrant type cavity, was estimated to be 33  $n\Omega$  at 150 MHz. The residual resistance measured for unpolished 5µ surfaces was enhanced by a factor of 10. In [30], the residual resistance at 136.7 MHz was found to be 4.7 n $\Omega$  for unpolished 9.1 $\mu$ electroplated lead in a fluoborate bath and to be independent of temperature from 1.5 to 4.5 K. In [31],  $R_{\text{RFS}}$  of a 0.3  $\mu$  thick lead film thermally evaporated onto sapphire (oil-free UHV system below 10-9 Torr, 99.9999% pure lead) corresponded to 62 n $\Omega$  if adjusted to 150 MHz and 4.2 K. The overall performance of real cavities is poorer than those results obtained with test samples. The table presents the current performance of low beta lead-plated RF structures employed at Stony Brook, Munich, Legnaro and Canberra. All cavities operate at frequency in the range from 150 to 170 MHz.

Table: Performance of lead-plated RF resonators

Institution/	d,	E <sub>acc</sub> ,	Γ,	Q at E <sub>acc</sub>	R <sub>s</sub> ,	Ref.
Cavity	μ	MV/m	Ω	$x10^{8}$	$n\Omega$	
SUNY/SLR	2	2.0	20	1.5	130	[1]
TRITRON	5	4.7	94	3.7	254	[28]
ALPI/QWR	2	2.0	32	2.0	160	[4]
Oxford/SLR	1	2.0	20	0.8	250	[34]
ANU/SLR	2	2.0	20	$2.0^{\text{a}}$	100	[34]

<sup>&</sup>lt;sup>a</sup> Design goal

Some possible sources of  $R_{\rm RES}$  in a cavity fabricated by plating PbSn onto copper are dielectric or magnetic contaminants, small particles of foreign normal metals, exposed spots of normal substrate from imperfections in the plating, areas of normal superconductor caused by trapped magnetic flux and phonon generation by interaction of RF fields with the lattice [32]. The brief

phenomenology of residual resistance of lead deposited onto copper is discussed below.

## 6.1 Technological losses

Technological losses relate to RF gaskets, beam cut-off tubes, pick-up and coupler holes. By using carbon thermometers, an estimate of loss distribution over an SLR was obtained in [33]. In the quadratic region, 2.4 MV/m, half of the losses are associated with the loop itself and half with the can structure and the indium joint between the loop and the can. In [35] thermometry measurements demonstrated that, in absence of field emission, only about 5% of the total 4.2 K losses occur on the resonator outer housing.

## 6.2 Dielectric and magnetic losses

In [36] the observed frequency dependence of the losses  $(\mathbf{Q} \sim \omega^2)$  could result from a thin layer of dielectric in the capacitive gap with frequency independent dielectric loss angle  $\delta$ . The magnitude of the losses for the highest Q would result from a surface dielectric film characterized by  $dtan \delta / K = 0.1$  Å, where K is the dielectric constant and d is the film thickness. The results of [37] indicate that all the samples from the lead cavities had a surface <sup>16</sup>O concentration that would correspond to a PbO thickness of about 100 Å. The samples had been stored to avoid exposure to atmospheric humidity and no correlation of <sup>16</sup>O concentration with age was seen. Halbritter in [38] shows that dielectric losses ~ωε"E<sup>2</sup> are no major cause for  $R_{RES}$ . Measurements on  $O_2$ ,  $N_2$ ,  $CO_2$  set an upper limit for  $ε''/ε^2$  of  $10^{-5}$ . In contrast, magnetic RF losses  $\sim ωμ''H^2$  are quite large reaching for  $O_2 \mu''/\mu \approx 10^{-2}$ . A condensed layer of about 1000 Å air can dominate the RF losses. During fabrication of a lead-plated cavity, complete exclusion of contaminated materials from solutions used in plating is not possible. In [30] several solvents were tested for cleanness to see which should be used as a final rinse after plating. Electronic grade acetone proved to be best, but even it left residue. The presence of such material on superconducting surface may contribute to dielectric and magnetic losses.

### 6.3 Surface roughness

Calculations in [37] did show that the local enhancement of the electric field due to microscopic surface roughness did not contribute to the dielectric losses. The increase in surface area from roughness does, however, increase RF losses of a superconductor if the penetration depth, 50 nm for lead at 150 MHz, is smaller than the surface structure. Halbritter [38] finds that surface roughness can greatly enhance phonon generation above that for a plane surface producing loss proportional to  $\omega^2$ . In a sufficiently rough surface, this mechanism can account for considerably

higher residual loss. Pierce [39] observed residual loss, which is interpreted as the result of an extreme form of surface roughness, namely, a whisker-like protrusion from the surface. Such a spike causes enhancement of the field and a local increase in dissipated power. Since the superconducting surface resistance increases rapidly with temperature and there is relatively poor thermal conductivity to the helium bath, there can be a thermal runaway that at high field RF level can keep the whisker and its immediate vicinity in the normal state.

# 6.4 Trapped magnetic flux

RF losses due to frozen magnetic flux  $R_{RES} \approx R_0 H_{dc}/H_c$ , where the field  $H_{dc}$  may be externally applied, caused by thermoelectric current or generated during high power pulsing. In contrast to GHz frequencies, at lower frequencies <0.5 GHz, fluxoid losses increase with RF field and temperature thus requiring magnetic shielding in low frequency cavities. Below  $H_c$ , the Meissner effect expels magnetic fields from the interior of single crystal when it becomes superconductor. The smallest impurity of the material can be sufficient to prevent Meissner effect [30]. Under such conditions 'frozen in' magnetic flux may be found and the superconductor surface has flux-free surface areas alternating with normal areas trough which the flux passes.

The cavity could generate its own magnetic fields by means of thermoelectric currents which flow between the lead plating and the copper structure in the presence of temperature gradient ~0.1 K/cm such as exist while the cavity is being cooled during transfer if liquid helium [39]. A period of time exists when part of the lead is superconducting and part is not because of temperature gradients. There would be thermoelectric current, which generates magnetic flux. Some of this flux could be trapped as the lead becomes entirely superconducting and location of this flux would depend on the details of the cooling process, which are different each time a cavity is cooled.

#### 6.5 Mechanical stress

No evidence of strain induced magnetic losses in electroplated lead has been found in [36]. The maximum strains induced have approached the elastic limits of the copper substrate without noticeably affecting the RF properties of the superconducting surface. The difference of contraction on cooling from room temperature to 4.2K may be effectively regarded as a 'thermal pressure'. The stress in lead film reaches 0.7 kbar from equations of Ref. [40]. Using the value  $dT_c/d\rho \approx -4 \cdot 10^{-5}$  K/bar [20a] for the lead film, along with calculated value of the stress, the broadening of the transition to be  $\Delta T \approx 0.035$  K.

### 7 RF TESTING

After assembling the resonator into the cryostat and pumping for 12 hours, the resonator is warmed up to 80°C for 150 hours. After this period the 77K heat shield is cooled while the resonator baking continues for an additional 12 hours. Then the resonator is cooled to 77K using gas phase N<sub>2</sub>. The RF is applied at 200 W and with 10-30% duty factor. For SLR, by choosing the appropriate power levels, duty factor and coupling one can condition away nearly all the 77K multipactor levels in 4-12 hours.

Lead surfaces acquire hydrocarbons at every stage of lead technology. This was confirmed by SIMS and ERDA analyses of lead film deposited onto copper substrate. During low-field multipacting, hydrocarbons decompose under electron impact and leave carbon patches on the surface which subsequently may become field-emission sites. Discharge treatment in a CO<sub>2</sub> atmosphere proposed in [41] to assure oxidation of carbon and hydrocarbons into gaseous compounds, which are removed by pumping. During the second step, discharge treatment in a hydrogen atmosphere, lead oxides are reduced to metal. Recently a Freon discharge surface-processing method [42] has been successful in eliminating multipactoring in all types of lead-copper resonators.

The Q is measured by decay time at low field with critical coupling. Usually the Q falls sharply at modest fields as a result of field emission. During 'power conditioning' the resonator is pulsed to the maximum attainable field at critical coupling. Then helium gas is introduced at 10<sup>-5</sup> Torr. It takes 30-60 minutes with duty factor up to 10% at 200W for this process until the maximum attainable field is not increasing. After conditioning the low field Q often has deteriorated. After warming SLR above critical temperature of lead and subsequent re-cooling the low field Q returns to near the originally measured value. The reduction in Q may result from flux trapped during highpower conditioning as hypothesized in [43]. The effect of conditioning can be seen in figure 9.

The curves show the Q versus accelerating field after conditioning. Q value can be used to infer the surface resistance  $R_{\rm s}$  through the general resonator relationship  $QR_{\rm s}{=}\Gamma.$  The geometric factor for this resonator, given by  $\Gamma{=}20~\Omega,$  and the  $Q{=}~1.0x10^{\rm s}$  measured at 2.0 MV/m (surface magnetic field of 220 Gauss), imply that  $R_{\rm s}=200~\rm n\Omega$  at 150 MHz and 4.2K. This is an order of magnitude too big to explain BCS theory and the observed RF losses in the resonator. In order to push the lead technology to its limit, a systematic investigation of the fundamental properties of lead as RF superconducting material with greater emphasis on high field performance is still needed.

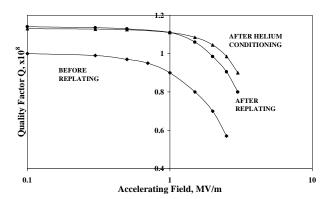


Figure 9. Performance of ANU SLR "300" before and after re-plating. After the helium conditioning the high field Q improved, but there was drop in the low field Q.

### **8 CONCLUSION**

Lead plating technology surveyed in this paper makes possible routine operation of the SLR at 3.5 MV/m at 6 Watts as showing by Noè [1a]. Cryostat refurbishment time is expected to be about one month. Only two-three days is surface preparation and plating time. Visual inspection of plated pieces is adequate. Cryostat refurbishment includes disassembling, assembling, cryostat upgrade and SLR frequency ageing. ANU LINAC upgrade comprising twelve SLRs in four cryostats will be completed during the year 2000.

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